ER-5670

N64-23521

cat. 19

Codel   NASA CR 56544

CO₂ REDUCTION UNIT DESIGNED

FOR

CONTINUOUS ZERO GRAVITY OPERATION

INTERIM REPORT

NASA HEADQUARTERS (OART-RBB)

Contract No. NASw-650

NOVEMBER 20, 1963

cr

TRW ELECTROMECHANICAL DIVISION
THOMPSON RAMO WOOLDRIDGE INC.
CLEVELAND, OHIO

2 3521

ABSTRACT

A carbon dioxide reduction unit has been designed and fabricated. Carbon dioxide is reduced by hydrogen to carbon and water, using flat iron plates as the catalyst. Carbon and water are continuously removed from the reaction gases. Product water is electrolized to oxygen and hydrogen. All components have been designed for operation in a zero-gravity environment.

Preliminary tests on the one-half man capacity unit were carried out at a system pressure of 2 psig. Reaction rates up to 900 cc/min of reaction gases were obtained at a reactor temperature of 1100°F. Carbon was successfully removed from the reactor during operation of the system.

author

TABLE OF CONTENTS

1.0 INTRODUCTION AND SUMMARY

This is an interim report covering the first six month of work carried out at Thompson Ramo Wooldridge Inc. under Contract NASw-650. This contract is for the design, fabrication, and testing of a Carbon Dioxide Reduction Unit capable of operating in a zero-gravity environment. The overall unit consists of a water electrolysis unit in addition to the CO_2 reduction reactor and its associated components.

The operation of the unit may be briefly described as follows. Carbon dioxide is reduced by hydrogen over an iron catalyst in the reduction reactor, producing water and solid carbon. The carbon is continuously removed from the reactor by a filtering system. Water which is produced is removed from the reaction gases and passed into the electrolysis unit where oxygen and hydrogen are evolved as gases. The oxygen produced is available for the cabin air, while hydrogen is added to the carbon dioxide and fed into the reactor. A CO_2 concentration unit is not included in the present contract coverage. Carbon dioxide reduction capacity of the unit was designed to be 1.2 pounds per day.

Unique design features included in the present carbon dioxide reduction unit are:
1. Method of continuous carbon removal
2. Porous plate condenser-separator used for water removal
3. Water electrolysis unit design.

In environmental control systems designed for use in closed cabins over extended periods, the oxygen regeneration unit is one of the most critical components yet to be developed. Long operation of an oxygen regeneration unit suitable for use in a closed system has not been demonstrated. The object of this program is to demonstrate such operation with the capability for continuous removal of carbon from the reduction reactor. Additionally, the development of a compact and efficient water electrolysis unit is required.

Design, fabrication, and preliminary testing of the CO_2 reduction unit has been satisfactorily completed. The reduction reactor has been operated at 150% of design capacity

1

and all other components have demonstrated satisfactory functional performance. The water electrolysis unit has been operated for 26 hours as an individual component, without being integrated with the rest of the reduction unit.

Testing of the CO_2 Reduction Unit will continue, to determine operating characteristics as a function of various parameters. During this testing phase, the electrolysis unit will again operate as an independent unit. After integration of the electrolysis unit into the CO_2 reduction unit, endurance testing will begin with operation at rated capacity.

As an extension of the present contract, it is recommended that the present CO_2 reduction unit be used as an experimental tool to determine design parameters for oxygen regeneration subsystems for actual use in space systems. Additionally, a follow on program is recommended wherein a completely integrated multi-man oxygen regeneration subsystem, including CO_2 concentration, be developed for testing at a NASA facility.

2.0   DESIGN DISCUSSION

The reduction of CO_2 with H_2 (over a catalyst) to form water and deposit carbon is one of the few methods sufficiently far along in development to be integrated into an oxygen regeneration subsystem for use in early manned space stations.   The reduction of CO_2 in the reduction unit may be summarized with the following equation:

$$CO_2 + 2H_2 \longrightarrow C + 2H_2O \tag{1}$$

Sufficient reports covering the background and chemical kinetics of this reaction exist [1, 2, 3, 4, 5]; a detailed discussion is therefore not warranted here.

2.1 CO_2 Reduction Unit

The CO_2 reduction unit can best be described by referring to the schematic flow diagram in Figure 2-1.   The major components of the unit are the reduction reactor, carbon filters, carbon collector, heat exchanger, condenser-separator, compressor, and electrolysis unit.   Carbon dioxide is supplied to the unit by bottle storage.   Thermal control consists of "simply-trol" control of reactor electrical heater (with manual override) and manual control of cooling water flow to the condenser-separator.

Bottled carbon dioxide is fed into the reduction reactor, where the reduction of carbon dioxide by hydrogen in high temperature catalyzed reactions gives the overall result indicated by the chemical reaction in equation (1).   Carbon which is produced in the reactor is continuously removed from the reactor by recirculation gases, then filtered from the gases by porous stainless steel filters.   As one filter approaches its capacity, the gas flow is diverted through the second filter; the first filter is then cleaned by back flow of gases through the carbon collector, causing the carbon to be deposited in the collector.   Back flow is accomplished by closing filter selector valves and opening carbon blow out valves associated with the loaded filter.   When these two valves have been properly positioned, the recycle gas is diverted through the carbon collector loop by closing the main carbon blow out valve on the control panel.

CO₂ REDUCTION UNIT SCHEMATIC

FIGURE 2-1

7

The recirculation gases (CH_4, CO, CO_2, H_2, and H_2O) after passing through the carbon filters enter the regenerative heat-exchanger and condenser-separator, where gases are cooled and the reaction water is removed. A filter is placed in the line before the condenser-separator to prevent any foreign particles from entering the pores in the condenser-separator. From the condenser-separator the recycle gases are compressed and fed through the catalyst drive motor chamber (motor cooling), then through the heat exchanger before being enriched with CO_2. Fresh H_2 feed is supplied to the recycle gases at the condenser-separator inlet. After addition of CO_2 feed, the entire gas stream enters the reactor. Methane and CO are not produced in the reactor beyond that amount required to maintain equilibrium. Once equilibrium is established in the reactor, no additional CH_4 or CO will be formed since these cases are not removed from the system.

Water from the condenser-separator is forced into a water accumulator. Provision for make-up water is included in the accumulator. A precision piston pump meters a set amount of water into the electrolysis unit. The feed rate is manually set. A pressure controller will maintain a balance between the water feed pressure and the gas output pressure. Any excess H_2 which is produced is vented by a needle valve. Power input to the unit is manually set and adjusted to provide a constant H_2 output from the cells. To prevent damage to the electrolysis unit if dehydration should some-how occur, a temperature limit control will turn off the power supply when the pre-set temperature is reached. Since the output H_2 and O_2 contain water vapor, the hydrogen is injected into the recycle gas stream at the condenser-separator inlet so that the excess moisture is not introduced into the reactor.

Gas analysis is accomplished by a gas chromatograph just prior to hydrogen injection. This analysis determines what adjustments are necessary in the feed gas mixture ratio.

Sufficient indicating instruments are provided on the unit to monitor temperatures, pressures, flow rates, and critical voltages and currents.

A bypass line and valve on the compressor provides for flow control of the recycle gas stream.

2.2 Component Design

A layout drawing of the CO_2 reduction unit, Figure 2-2, shows the location of the major components. A discussion of the design of each of these components follows.

2.2.1 Reduction Reactor

The reduction reactor is the heart of the CO_2 reduction unit; associated with it are the major problems of the unit. These are:

1. Catalyst configuration
2. Gas confinement
3. High temperatures and materials compatibility
4. Carbon removal

Catalyst configuration must be determined first since this controls the reactor size and the basic reaction rates attainable with the reactor. Figure 2-3 presents a cutaway view of the reactor showing details of construction. Where possible all structural pieces of the reactor were fabricated with 316 type stainless steel.

2.2.1.1 Catalyst Configuration

Figure 2-3 shows that the catalyst material required for the reduction reaction has the form of a stack of evenly spaced disks. These disks are fabricated with low carbon steel sheets. A determination of the spacing between plates and area required to achieve satisfactory reaction rates was determined during TRW's experimental program prior to working on this contract. It was determined that a minimum spacing of 0.25 inche⸱ should be used with the flat plate configuration for optimum utilization of catalyst surface area. The average reaction rate per unit area was determined to be 0.5 cc/min per cm^2 of catalyst. Thus, for a reaction rate of 600 cc of $2H_2:CO_2$ mixture per minute the catalyst surface area must be at least 1200 square centimeters. This area requirement is satisfied by eleven disks of 5.5 inch diameter with a 0.75 inch
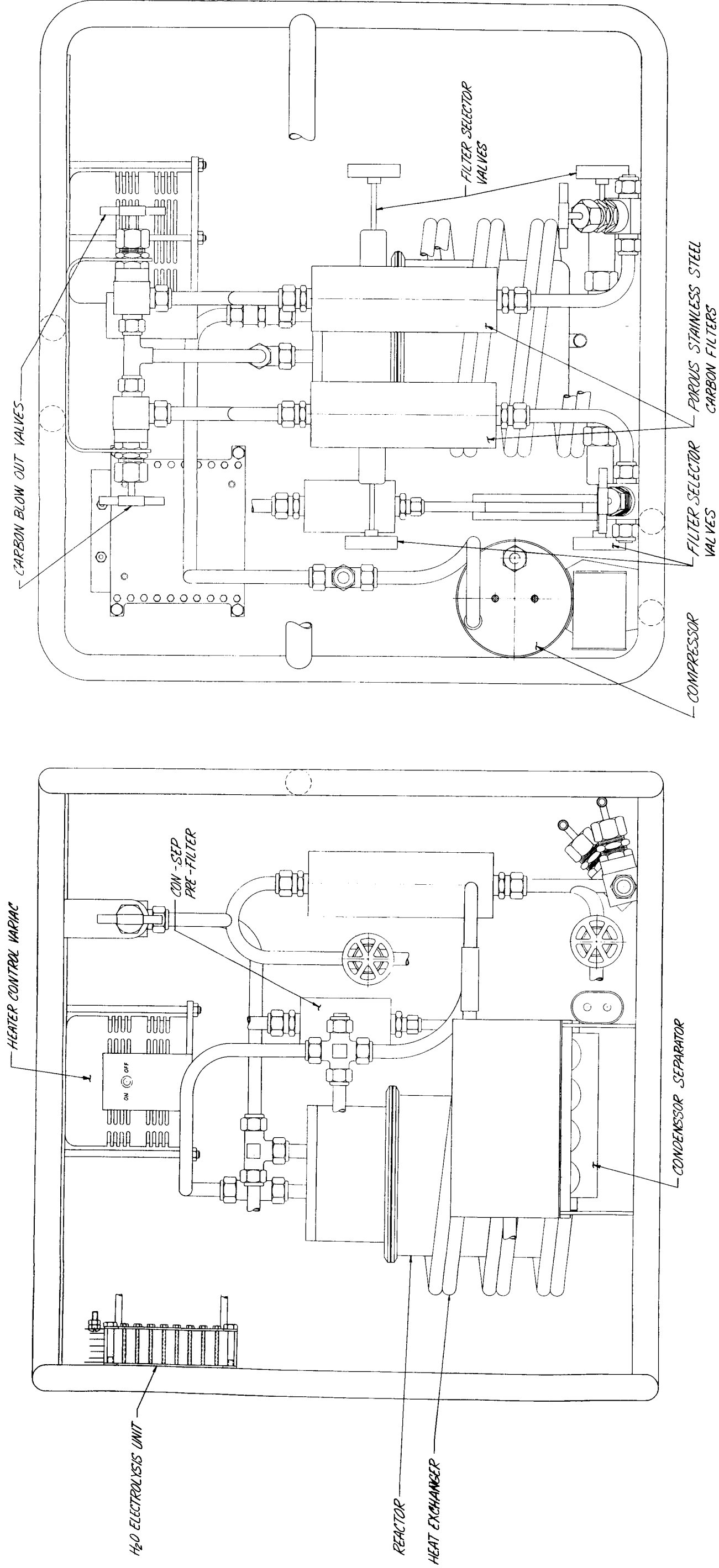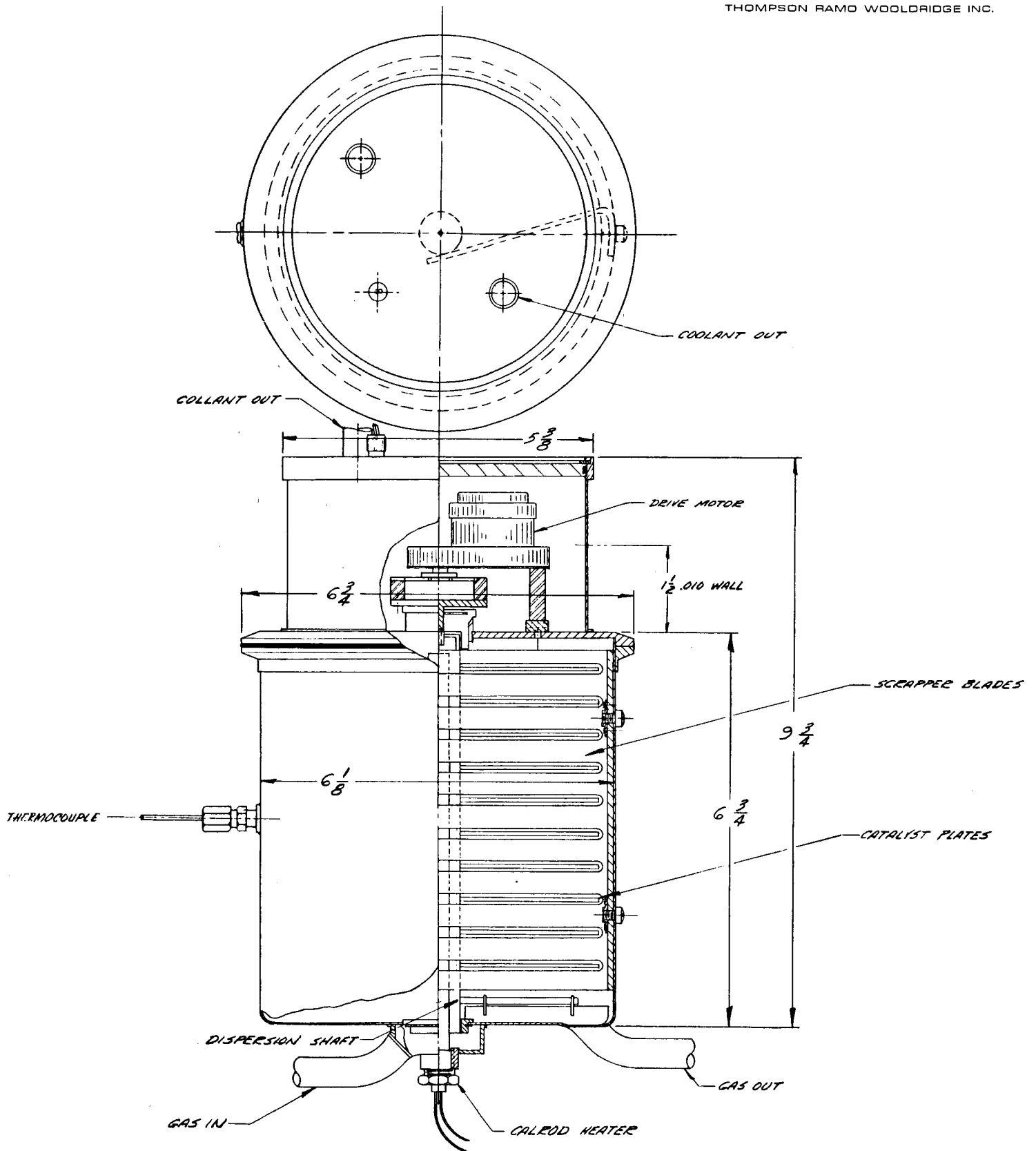
CARBON BLOW OUT VALVES

FILTER SELECTOR VALVES

POROUS STAINLESS STEEL CARBON FILTERS

FILTER SELECTOR VALVES

COMPRESSOR

HEATER CONTROL VARIAC

CON-SEP PRE-FILTER

CONDENSSOR SEPARATOR

H₂O ELECTROLYSIS UNIT

REACTOR

HEAT EXCHANGER

FIGURE 2-2

7

COOLANT OUT

COLLANT OUT

$5\frac{3}{8}$

DRIVE MOTOR

$6\frac{3}{4}$

$1\frac{1}{2}$ .010 WALL

SCRAPPER BLADES

$9\frac{3}{4}$

$6\frac{1}{8}$

$6\frac{3}{4}$

THERMOCOUPLE

CATALYST PLATES

DISPERSION SHAFT

GAS OUT

GAS IN

CALROD HEATER

REACTOR LAYOUT

TAPCO GROUP

818249

FIGURE 2-3

diameter center hole for mounting. The flow of reaction gases is outward from the central mounting tube. A theoretical analysis performed by TRW indicates that the cylindrical configuration as described (outward flow of reaction gases over circular catalyst plates) will give excellent reaction rates per unit area of catalyst. A spacing of 0.50 inches between plates was allowed to provide spacing for carbon removal fingers and a carbon deposit layer. Thus, disk diameter, total number of disks (area required), and disk spacing determines the reactor envelope.

The specific reaction rates given above are for a reaction temperature of 1200°F, condenser temperature of 60°F, and a recycle flow rate of 10 liters/min.

The catalyst disks are attached to a hollow tube which is continuously rotated by a drive motor mounted on top of the reactor. A high temperature electrical heater is mounted up the center of the mounting tube. Reaction gases enter the reactor through holes in the mounting tube placed between the stacked disks. Figure 2-4 presents a photograph showing the catalyst stack and the catalyst drive motor.

With this catalyst arrangement and the reaction conditions stated, a carbon deposition to iron consumption ratio of approximately 10 is expected.

2.2.1.2   Reaction Gas Confinement

The problem of absolute confinement of hazardous reaction gases is compounded by the high operating temperature of the reactor. Two rotating bearings are required in the present design. The bottom bearing is not a problem since a fitting could be welded onto the reactor completely sealing this bearing. The bearing through the top of the reactor could not be totally enclosed since a drive motor is attached on this end of the catalyst mounting tube. Initially, it was planned to use a carbon rotating seal at this point to prevent outward flow of reaction gases. This seal, however, did not give the required performance. To overcome this problem, the drive motor and bearing was completely enclosed by a welded housing.

CATALYST DRIVE MOTOR

REACTOR TOP

IRON CATALYST DISKS
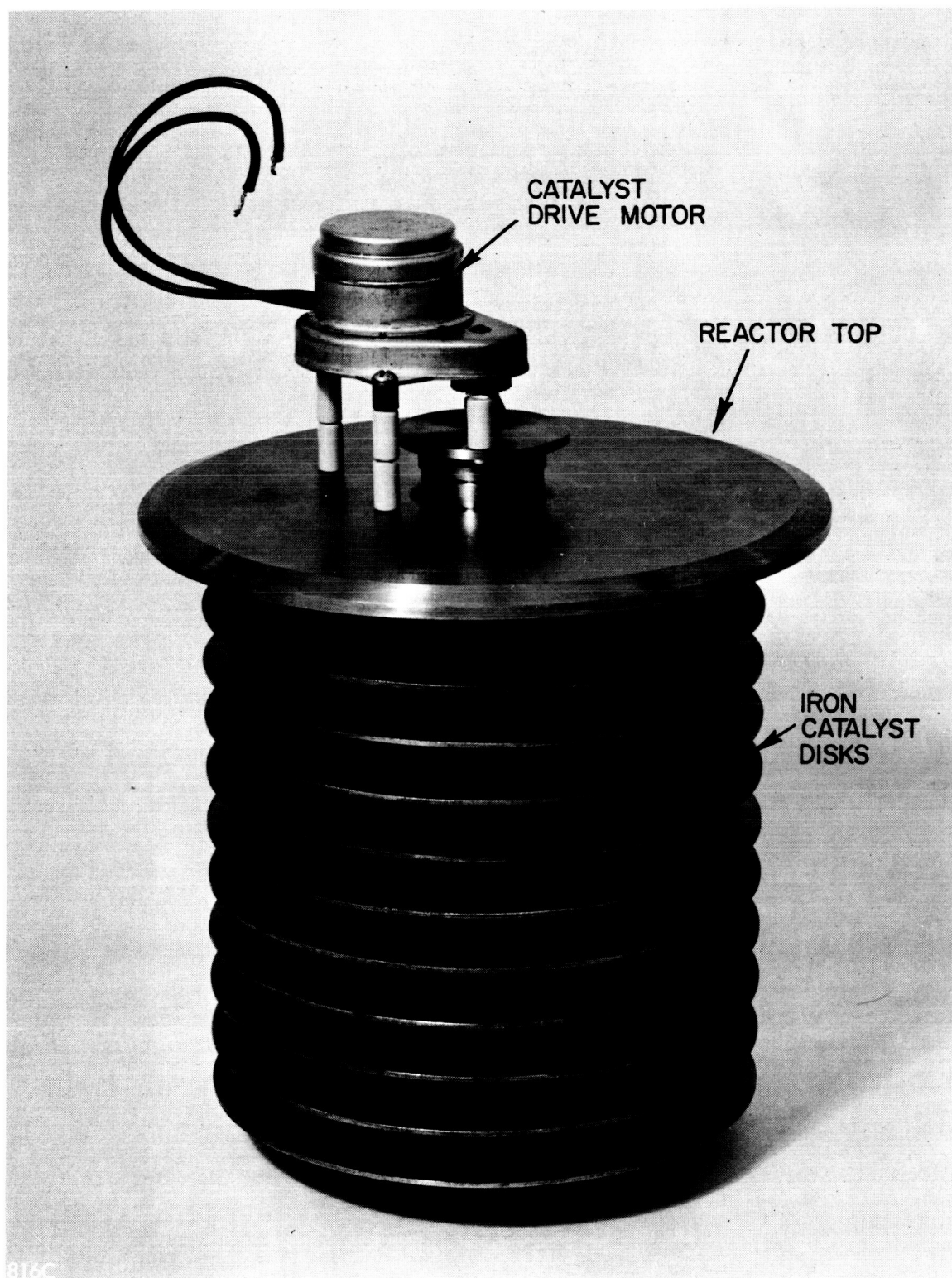
CATALYST ASSEMBLY AND DRIVE MOTOR   FIGURE 2-4

It was desired that the reactor have a top assembly which could be easily removed for inspection of the catalyst assembly. This was to be provided by a metal O-ring seal and quick release clamp arrangement. Adequate sealing was not obtained and it was decided that the reactor should be welded shut. This bead weld can be opened if inspection of the reactor interior becomes necessary. Figure 2-5 shows a view of the reactor incorporating the original design features.

Diffusion of hydrogen through the reactor vessel walls was calculated to be approximately 0.2 cc per hour for the oxidized stainless wall. Hydrogen diffusion was therefore not deemed to be a problem.

2.2.1.3 Thermal Considerations

The reduction reaction (equation (1)) is an exothermic process. However, the energy liberated by the reaction is less than the energy lost by cooling the reaction gases prior to water removal and by losses through the thermal insulation and along the metal tubing which connects the reactor to the rest of the system. The net energy deficiency, considering the worst possible conditions, amounts to approximately 180 watts. These conditions are:

1. Recycle Ratio - 30 (moles recycle bases/moles feed gases)
2. Temperature of Gases Entering Reactor - 600°F
3. Insulation Layer Thickness - 2 inches (K = 0.55)
4. Energy Loss by Conduction Equal to 25% of Losses Through Insulation

This energy deficiency will decrease to 75 watts if the recycle ratio is decreased to 10, inlet gas temperature increased to 850°F, and insulation thickness increased to 3 inches.

An electrical heater having a capacity of 400 watts is provided to reduce the time required to bring the reduction unit up to reaction temperature. Figure 2-3 shows that the heater, enclosed in an incoloy sheath, is mounted in the center of the rotating catalyst assembly. A maximum sheath temperature of 1750°F can be tolerated by the heater. A manually controlled variac controls heater power, while an Assembly
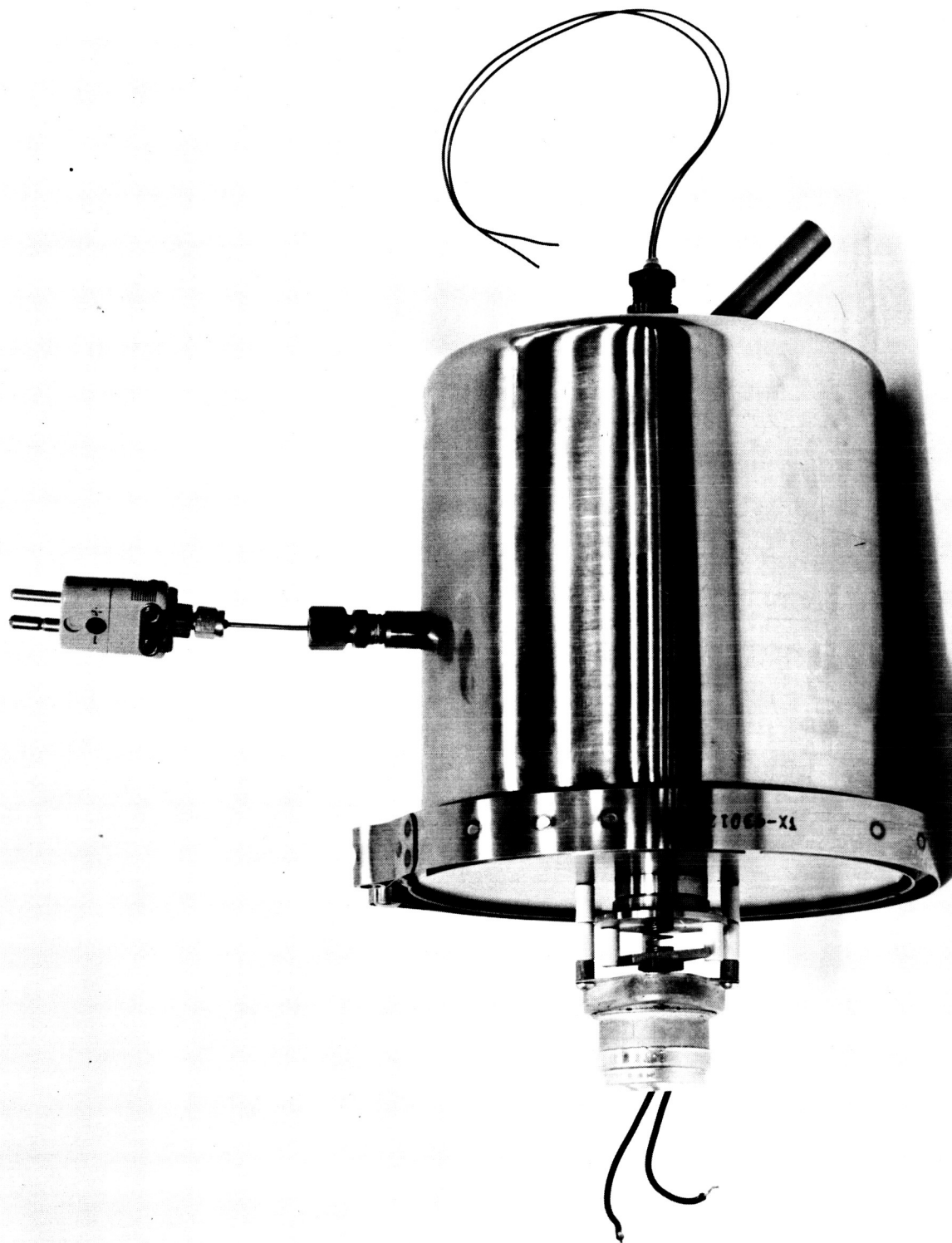
REDUCTION REACTOR - INITIAL DESIGN

FIGURE 2-5

Products Inc. "Temp-Tendor" and thermocouple controls power "on-off" to the variac for temperature limit control.

Thermal insulation used on the reactor was "J-M" Thermoflex and "J-M" Micro-Fibers Felt Type E. This material has a K value of approximately 0.55 at a mean temperature of 700°F.
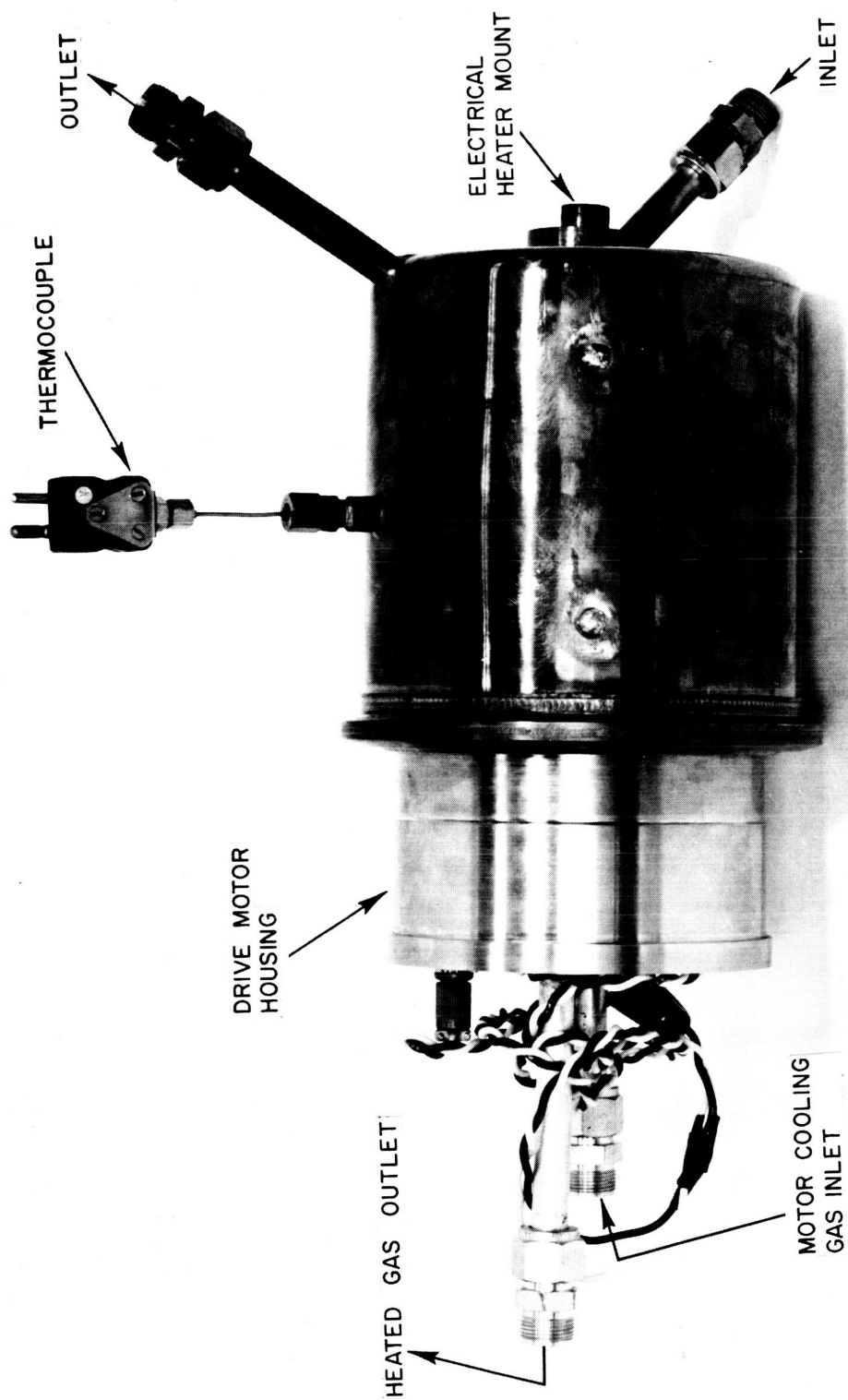
The enclosure on top of the reactor to prevent leakage of gases through the rotating seal caused a severe thermal problem for the catalyst drive motor. Heat transfer to the motor chamber is minimized by placing layers of insulating material between the motor and hot reactor surfaces. A thin walled section is machined on the enclosure case adjacent to the reactor top to reduce heat transfer up the housing sidewall. The motor mounts are heat-treated lava. Cool reaction gases are passed through the housing and over the motor to maintain a suitable operating temperature. When the reactor temperature is 1200°F, the motor chamber is approximately 120°F.

Figure 2-6 shows the modified reactor with motor enclosure after preliminary testing. The difference in oxide coating thickness along the housing wall gives an indication of the temperature along the housing wall.

2.2.1.4   Carbon Removal

Continuous carbon removal is required if a practical reduction unit is to be developed. The method adopted in the present unit depends on easily achieved mechanical processes. Carbon is deposited on the slowly rotating catalytic disks, which are kept free of thick carbon deposits by the rotation past a bank of carbon removal fingers. The carbon which is removed from the disks is carried from the reactor by the normal gas flow over the disks. This stream of gases and carbon particles passes through a porous stainless steel filter where the carbon is removed.

Since the carbon buildup can be tolerated on the catalyst surface, only the loose carbon above the surface must be removed from the disk. In this mode of operation, the carbon removers do not actually have to touch the surface of the moving disks and thereby

OUTLET

ELECTRICAL
HEATER MOUNT

INLET

THERMOCOUPLE

DRIVE MOTOR
HOUSING

HEATED GAS OUTLET

MOTOR COOLING
GAS INLET

MODIFIED REDUCTION REACTOR - AFTER PRELIMINARY TESTING

884C

FIGURE 2-6

14

decrease the problem of wear. Figure 2-7 shows an interior view of the reactor with the carbon removal fingers in place.
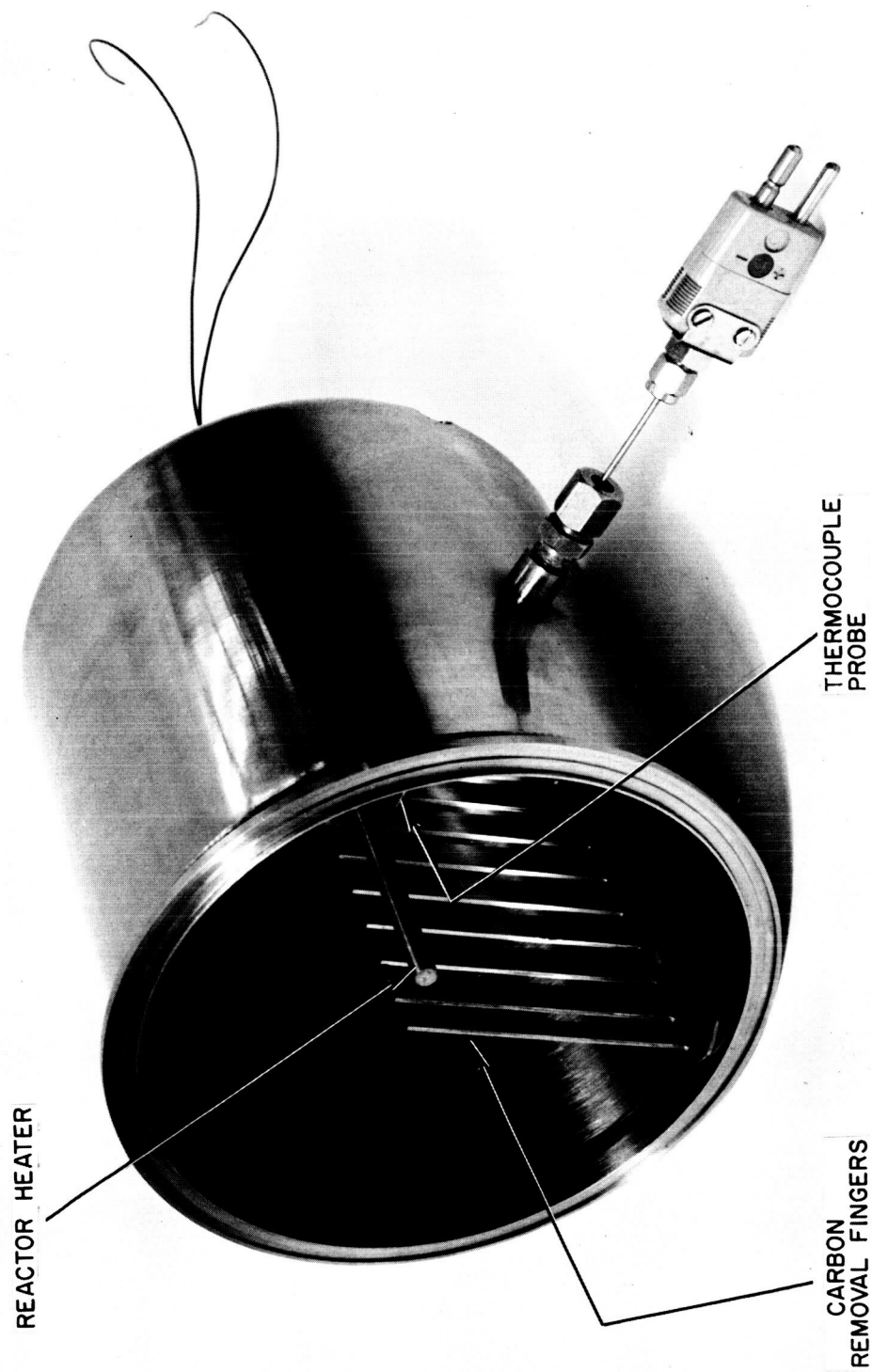
2.2.2 Heat Exchanger

The heat exchanger for the CO_2 reduction unit is required in order to recover the thermal energy of the gases in the recycle stream which must be removed in order to condense the reactant product water. The driving force for the exchanger is the sensible entropy difference which is generated as a result of energy removal in the condenser-separator unit inserted in the line between the hot and cold loops of the heat exchanger.

A counterflow type of heat exchanger is used. Geometrically, this consists of two side-by-side tubes, carrying the hot and cold flows. The energy flux occurs across the common junction of these tubes. The most efficient design would result if tubes of square cross section were used with the major thermal energy transport occurring across a plane surface. However, because of fabrication ease, tubes of circular cross section were used. An empirical law indicates that an exchanger built of circular tubes will have 85% of the effectiveness of a similar one built of tubes with square cross sectional shape. Using 0.5 copper tubing brazed together with brass it was calculated that a heat exchanger 4.0 feet long would be required. Preliminary testing indicated that this length was not adequate. Using this preliminary test data, the new length was calculated to be approximately 6.0 feet. Testing indicated an effectiveness of 0.955, which is satisfactory. The temperature into the condenser-separator should be 160°F.

2.2.3 Carbon Filters

Two types of filters are required to completely remove the product carbon from the reduction process stream: a high temperature regenerable filter and a large carbon collection chamber and filter (low temperature). The carbon collection chamber has sufficient capacity for three weeks continuous operation. The bulk carbon can be removed from this container while isolated from the remainder of the system. This low temperature filter chamber is fabricated with clear acrylic plastic with a 0.5 inch thick felt pad for the filter element.

THERMOCOUPLE
PROBE

REACTOR HEATER

CARBON
REMOVAL FINGERS

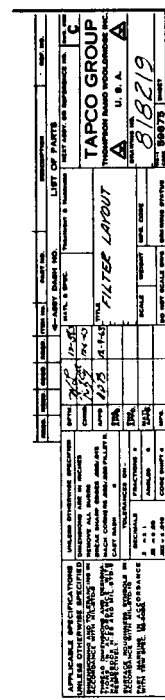REDUCTION REACTOR - INTERIOR

FIGURE 2-7

16

The high temperature carbon filters are placed in the system between the reactor and heat exchanger. These filters are fabricated of two concentric cylinders. The inner cylinder is a porous stainless steel with a solid disk welded to one end, while the outer cylinder is solid 316 stainless steel. Figure 2-8 shows a cross-sectional layout of the filter. The porous stainless steel filter element is a Pall Corporation element part No. C-12-6, Grade G. Mean pore size is 10 microns, giving absolute filtration of particles 1.8 microns or larger from gas streams. For 1/16" thick material, the pressure drop for air flow at 0.5 CFM is 0.08 psi when the filter is empty. Pressure drop through a filter 80% filled with carbon is expected to be 0.3 psi. These numbers were determined by actual testing of gas flow through beds of carbon generated by various TRW CO_2 reduction reactors. Each filter will be filled with carbon and emptied by back flow into the carbon collector once a day.

2.2.4 Condenser-Separator

The condenser-separator unit performs the function of removing the water from the recirculating gas stream and providing dry gas to be fed back into the reactor. To accomplish this end it is necessary that additional sensible heat be removed from the gas stream over and above that removed in the regenerative heat exchanger. The temperature level must be reduced to that corresponding to the saturation temperature of the gases; then the latent heat corresponding to the required amount of water to be removed must also be removed from the stream.

Figure 2-9 shows an exploded view of the condenser-separator. Water condenses on a cold plate as the recirculation gas flows through the unit. The cold plate upon which the condensation occurs is in reality a porous plate of stainless steel covered with a sheet of asbestos 6 mils thick. The plate has a 10-20% porosity and a pore size of 1 to 2 microns. The H_2O upon condensing on the porous surface is immediately drawn through the pores by means of the capillary forces. The water collects in the passages machined on the surface of the copper back-up plate.

FIGURE 2-8

CONDENSER-SEPARATOR ASSEMBLY

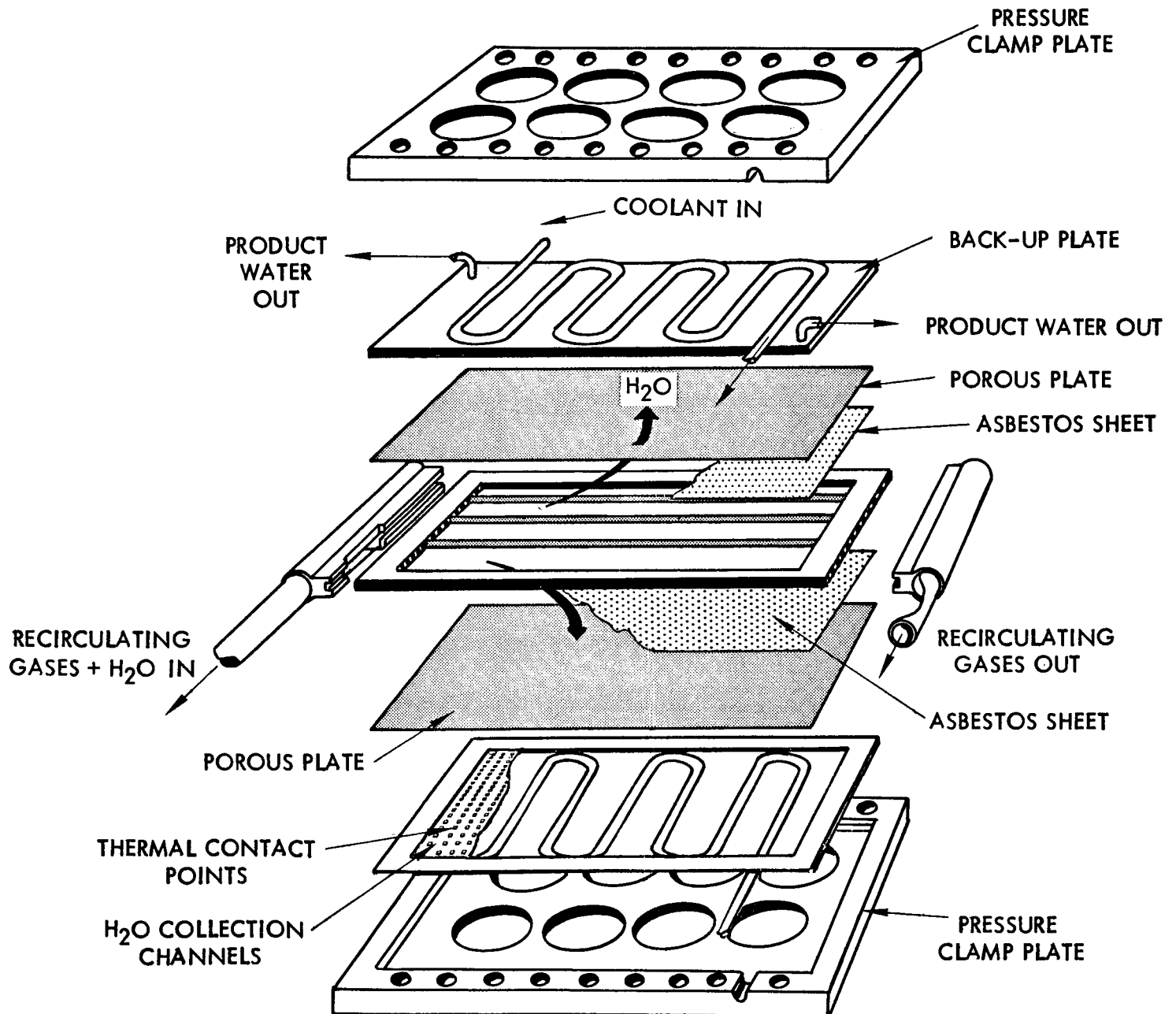PRESSURE CLAMP PLATE

COOLANT IN

PRODUCT WATER OUT

BACK-UP PLATE

PRODUCT WATER OUT

H_2O

POROUS PLATE

ASBESTOS SHEET

RECIRCULATING GASES + H_2O IN

RECIRCULATING GASES OUT

ASBESTOS SHEET

POROUS PLATE

THERMAL CONTACT POINTS

H_2O COLLECTION CHANNELS

PRESSURE CLAMP PLATE

FIGURE 2-9

The size of the condenser-separator is determined by heat transfer requirements through the raised stubs on the copper back-up since more than adequate area is available for flow of condensed water through the pores.

The porous stainless plate used in this condenser-separator can resist a capillary pressure of 5 psi. To prevent passage of gas through the plate should minute flaws develop, the asbestos layer was added. The asbestos is capable of resisting a capillary pressure of at least 15 psi.

Cooling is provided by cold water flowing through the cooling coils brazed on the backs of the back-up plates. Coolant temperature will be maintained at about 45°F in order to obtain a recycle outlet temperature of 50°F. Cooling water flow rate is manually controlled.

This type of condenser-separator produces very slight pressure drops in the gas flow through the unit, while providing recovered water at a positive pressure.
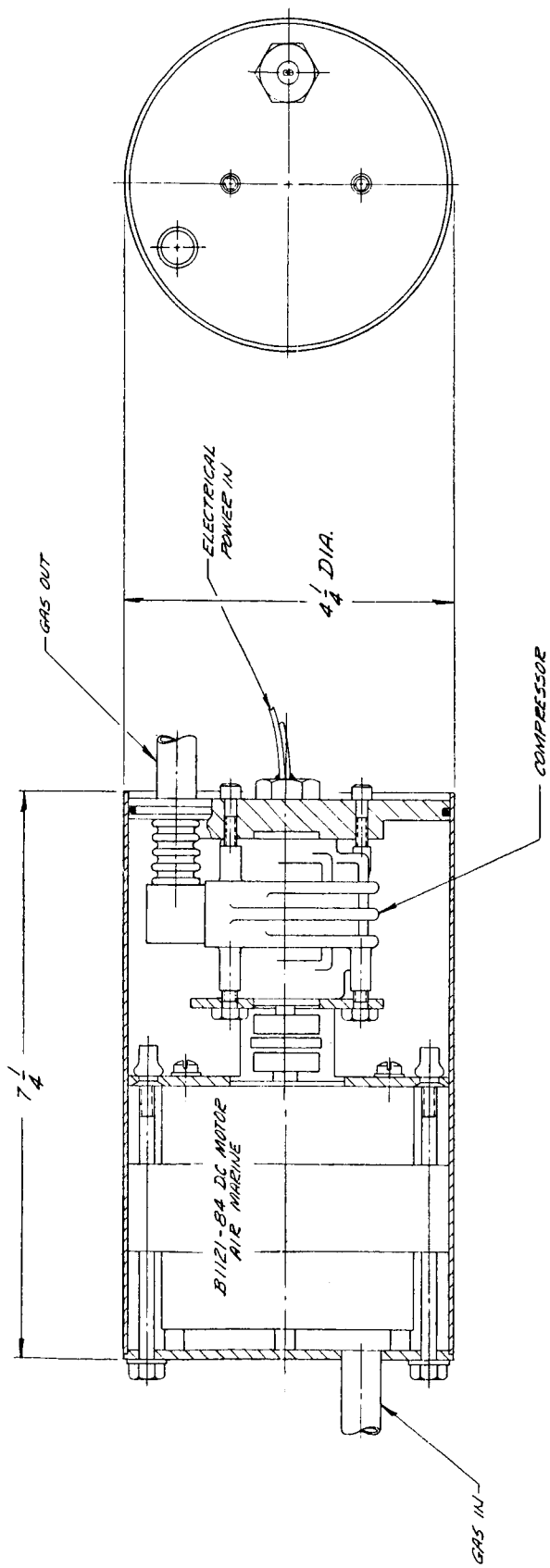
Pressure plates are used to hold the pieces together, with RTV silicone rubber used as a sealant and bond between the porous surfaces, the main frame, and the copper back-up plates.

2.2.5 Compressor

Blow requirements for the CO_2 reduction unit are for relatively low flow rates coupled with a significant pressure rise through the blower. The design point calls for 0.5 CFM at a pressure drop through the system varying from 2-4 psi.

Variations in pressure drop is due to collection of carbon in the filter chambers.

These gas flow requirements are satisfied by a carbon vane compressor driven by a constant speed motor. Both motor and compressor are sealed in a single aluminum housing to prevent leakage of gases from the compressor shaft seals. Figure 2-10 presents a layout drawing of this compressor. Recycle gases from the condenser-separator (50°F) pass over the motor into the compressor inlet. Outlet temperature

20

GAS OUT

ELECTRICAL
POWER IN

$4\frac{1}{4}$ DIA.

COMPRESSOR

$7\frac{1}{4}$

B1121-84 DC MOTOR
AIR MARINE

GAS IN

FIGURE 2-10

TAPCO GROUP
COMPRESSOR LAYOUT

will be approximately 100°F. Motor cooling is accomplished by recycle gas flow through the blower unit.

The motor used is an Air Marine A. C. 60 cycle Induction motor, Model No. B 1121-84 using 98 watts electrical power while running at 3300 rpm. The compressor is Gast Model No. 1030 oil-less carbon vane air compressor.

2.2.6 Electrolysis Unit

The design and construction of an efficient electrolysis unit for gravity free environment depends on the use of the best possible electrodes and on a suitable feeding system for water. The electrode material used in this model is a product of American Cyanamid. It consists of a 100-mesh nickel screen coated with platinum black. Teflon dispersion is used as a binder.

With the electrode material fixed, the next step was to choose a feeding wick. Because of the presence of KOH, an inert material is a necessity. For the conditions of cell operation, polypropylene is the best readily available material. While it does not wet easily, once it is wet it can act as a good wick. The material used in the cell is a woven filter cloth with a nap on one side. It was selected not only because of its chemical inertness but also because of its ability to transport water.

The transport properties of the various wicking materials were determined by placing them between two acylic plates which were marked at 1, 2, and 3 inch intervals. The rate at which KOH as water solution was transported from bottom to top determined the material to be used. However, since the rate slows down as the height increases, the electrolysis cell was designed to minimize the maximum height from the reservoir to the top of the cell. This will aid the operation of the cell at 1g. At zero g this will no longer apply and almost any wick might do.
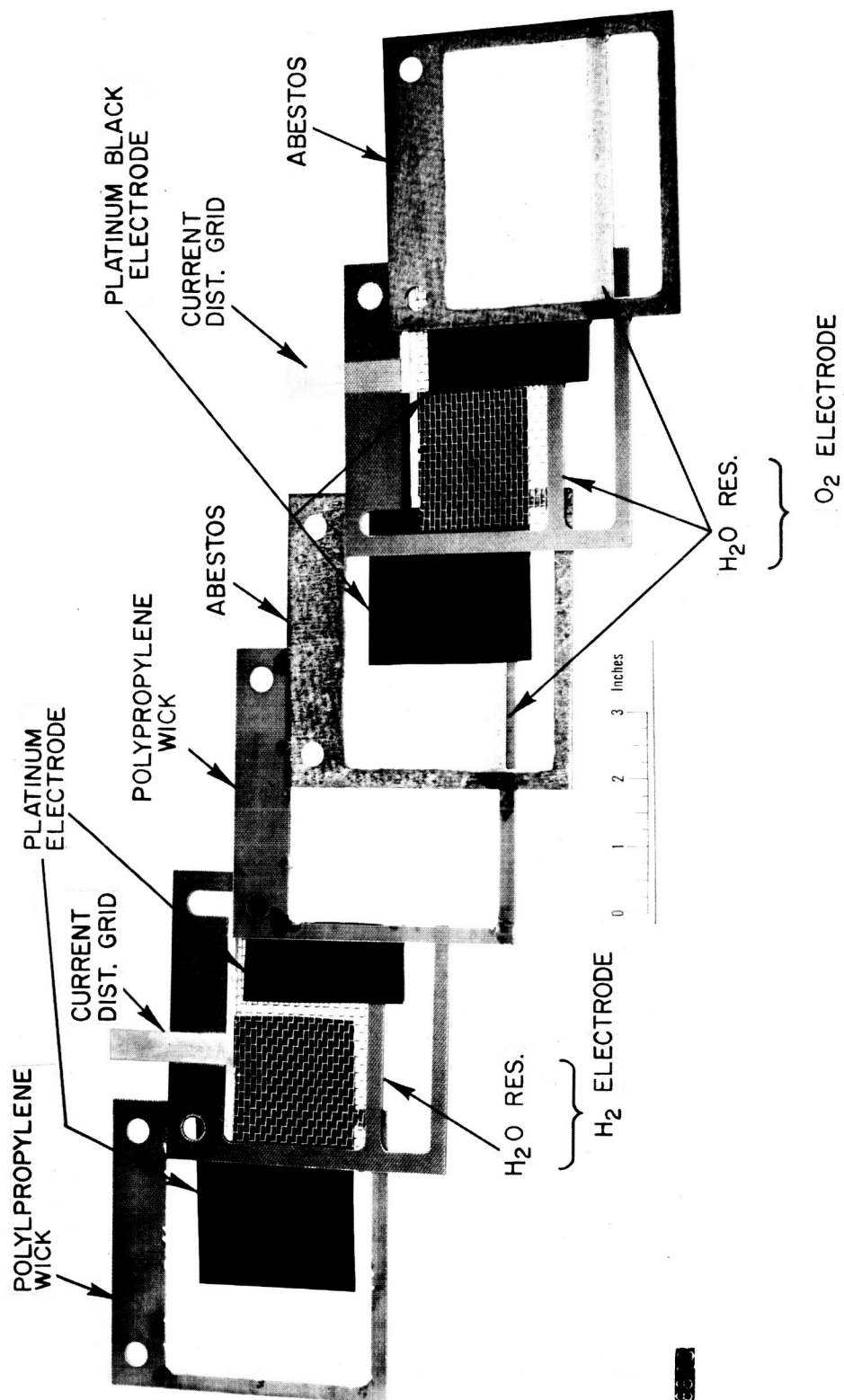
In addition to the wick and electrodes, an asbestos diaphragm is used. This prevents the mixing of the gases. The particular material used is a special 15-mil electrolysis cell material that withstands 15 psi differential gas pressure when wet.

The power requirements for the cell are those required by a half-man. Normally, the oxygen required by one man is produced by a current of 100-125 amperes, this figure depending on the source of the information. For a conservative design, the 125 ampere figure was chosen, so the cell is designed for 65 amperes with a current density of 100 ASF. The result is a cell which could operate at much higher currents if necessary. Because of the type of cell geometry, this conservative design requires very little extra space.

The materials of construction, in addition to those already mentioned, are epoxy-glass fiber board, monel screening, and copper. All of the metal items were plated with a nickel flash after fabrication to protect the copper and monel from the KOH. Figure 2-11 shows how the parts are assembled to form individual cells of the unit. These parts are bonded to each other with an epoxy cement which is alkali resistant. The entire assembly is capped to two end plates. One of these is a piece of epoxy-glass fiber board, the other is an assembly of tubes silver brazed to a copper sheet and then nickel plated. It is then covered with an insulating epoxy-glass fiber board. The entire assembly is held between two bowed steel plates. When the side bolts are tightened until the plates are flat, there is enough pressure against the assembly to allow safe operation of the unit at low pressures. The total assembly is then potted with epoxy to give added strength. Figure 2-12 is a drawing of the completed assembly.

The internal construction of the electrolysis cell is such that the pressures of the H_2 gas and water feed must be equal. Thus a method of increasing feed water pressure and output gases must be used to prevent electrolyte blow out or feed water blow back. Water feed into the electrolysis unit will be accomplished by a Harvard Apparatus Co., Lambda Pump capable of accurate control of flow rates. The flow rate is capable of being controlled in steps of 0.006 cc per minute. The approximate flow rate required is 0.366 cc per minute. Control is manual.

Pressure balance between pump and gas output will be maintained by a Honeywell "Cellmaster" pressure control. Tests have shown that with a "Cellmaster" and a

PLATINUM BLACK ELECTRODE

CURRENT DIST. GRID

ABESTOS

ABESTOS

POLYPROPYLENE WICK

PLATINUM ELECTRODE

CURRENT DIST. GRID

POLYLPROPYLENE WICK

H_2O RES.

O_2 ELECTRODE

H_2O RES.

H_2 ELECTRODE

inches

0   1   2   3

H_2O ELECTROLYSIS CELL COMPONENTS

FIGURE 2-11

24
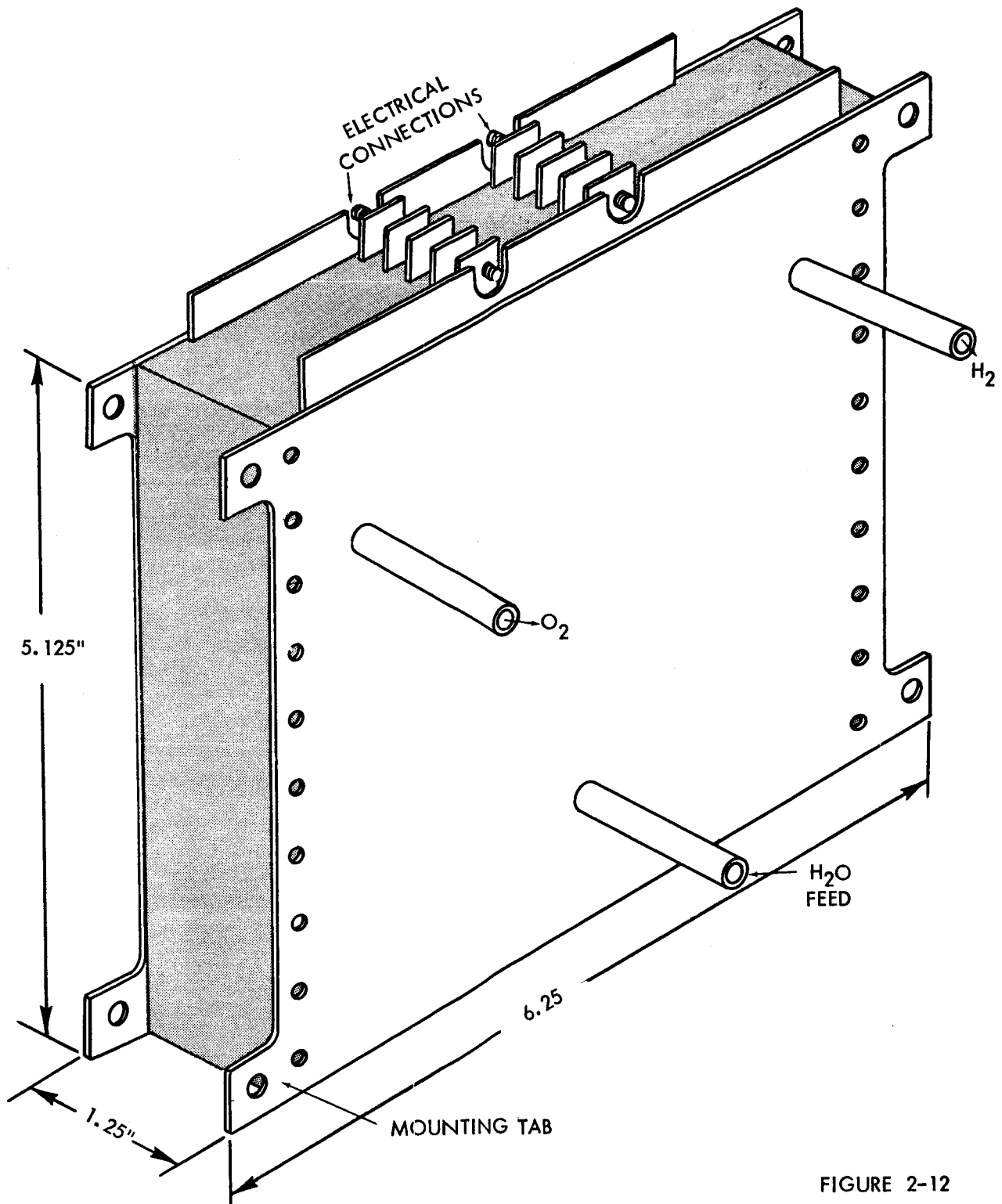
ELECTROLYSIS UNIT

FIGURE 2-12

simulated electrolysis unit, the pressure difference between feed water, O_2 gas, and H_2 gas was maintained at less than 0.25 inches of H_2O, while water pressure was varied from 0-15 psig. Manual pumping rate control will control feed water pressure.

2.2.7 Miscellaneous Components

A number of minor components are extremely important to proper operation of the unit. Without these components, failure of the unit would result after a relatively short period of operation.

Just prior to the condenser-separator is a small filter of fiber glass filling to remove any particulate matter which might enter the pores in the condenser-separator, and thus clog the plates and prevent the passage of water. The amount of foreign matter which enters this filter during normal operation is very small, so the filter will not require cleaning during the planned period of operation.

A moisture trap placed in the recycle flow line at the condenser-separator outlet removes the small amount of water which is not removed in the separator. This prevents slugs of water from being drawn out of the flow line and into the compressor. Trapped water is removed by a valve in the bottom of the trap.

Output water is collected in a small accumulator from which the Lambda Pump will draw water for the electrolysis unit.

Gas composition analysis is performed using a Beckman GC-1 Gas Chromatograph with molecular sieve and silica gel columns.

3.0   PRELIMINARY TESTING

Testing of components prior to assembly into the CO_2 reduction unit was necessary in order to insure adequacy of component design and to check purchased components for specified performance.   Upon completion of the unit testing and prior to initiation of the endurance run, it is necessary to determine what modifications are required for proper operation and what operating characteristics will yield optimum performance.

3.1   Component Testing

All components were pressure checked and subjected to the thermal and gaseous environment expected during actual operation.   Additionally, the following functional tests were performed.

3.1.1   Reactor

The most important functional test for the reactor was the determination of the reaction rate for the catalyst configuration in the reactor.   A sufficiently high rate must be obtained or the reactor is useless in that configuration.   Figure 3-1 presents a plot of the reaction rate and reactor temperature as a function of running time.   The present unit was designed for a nominal reaction rate of 600 cc/min °F $2H_2:CO_2$ gas mixture at a reactor temperature of 1100°F.   Figure 3-1 shows that a steady rate of 900 cc/min was achieved at 1100°F reactor temperature.

It shoul be noted that the condenser temperature was approximately 72°F or 20°F higher than the planned condensing temperature.   A lower condensing temperature favors a higher reaction rate.   Recycle gas flow rate was 13 liters/min.   Recycle gas flow rate for the planned testing will be approximately 20 liters/min.   Higher recycle gas flow rates favor higher reaction rates.   It was thus shown by this preliminary testing that the reactor is designed so that adequate performance can be expected.

During preliminarh reactor testing, two types of electrical cartridge heaters failed below their rated operating temperatures.   A source was located for heaters capable of operating in a 1750°F environment.

RECYCLE GAS COMPOSITION

N_2	-	12%
CH_4	-	4.5%
CO	-	23.0%
CO_2	-	24.0%
H_2	-	36.5%

PRELIMINARY REACTOR TEST
RECYCLE PARTS – 13 LITER/MIN
CONDENSER TEMPERATURE – 74°F
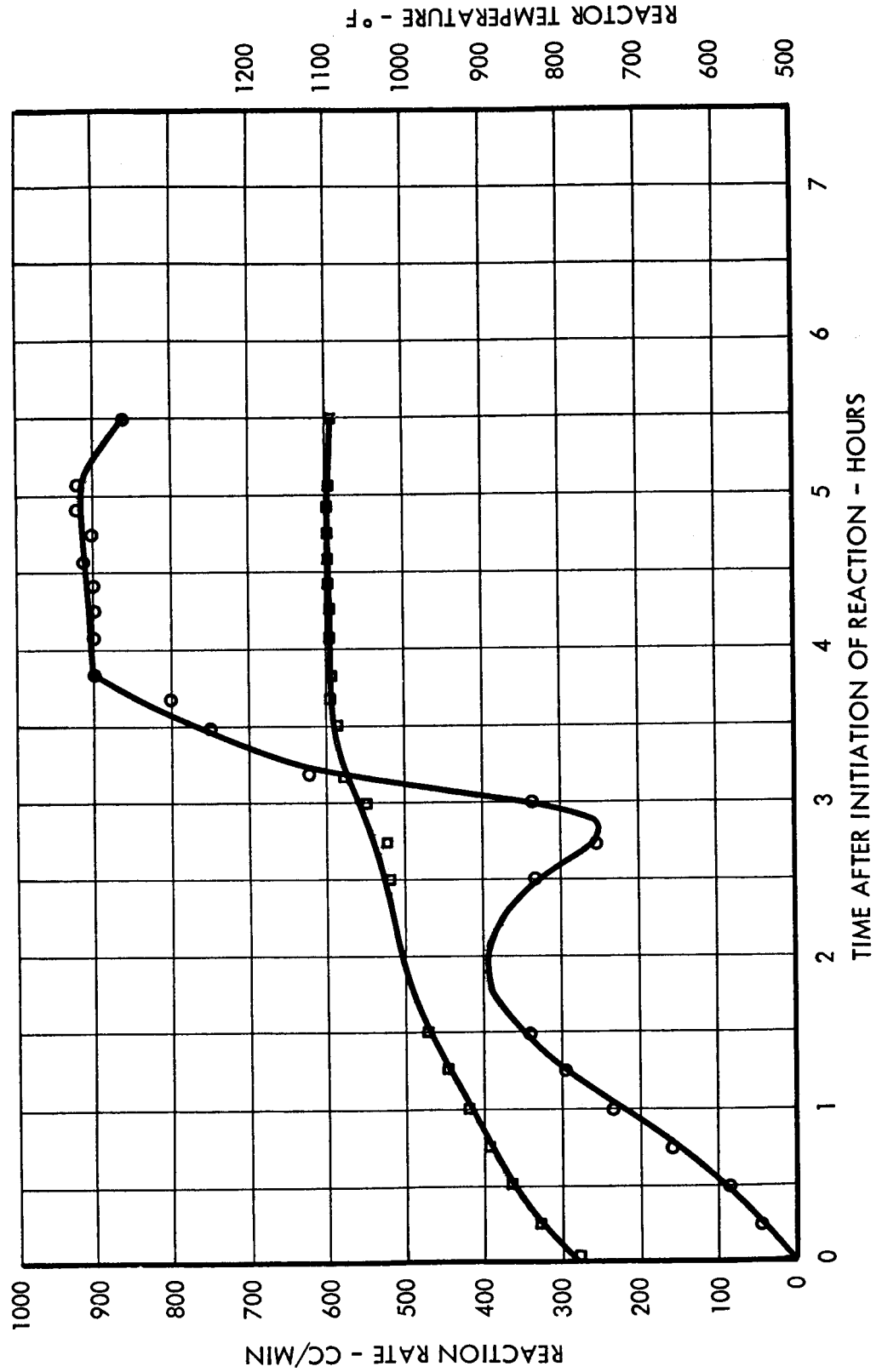
FIGURE 3-1

28

3.1.2 Heat Exchanger

Using a mixture of $2H_2:CO_2$ gas and an electrical heater to simulate the reactor, the heat exchanger was tested for pressure drop at rated flow and for outlet temperature on cold side. The first heat exchanger, four feet in length, had a pressure drop of 0.13 psi and an outlet temperature of 280°F when equilibrium was reached. This heat exchanger had an effectiveness of 83.5%. Since this performance was not satisfactory, a new heat exchanger was designed, fabricated, and tested. With this six foot long heat exchanger, a pressure drop of 0.18 psi was measured along with the following temperatures:

1. Hot side inlet - 1220°F
2. Cold side outlet - 144°F
3. Cold side inlet - 92°F

Heat exchanger effectiveness was calculated to be 95%. This performance was deemed to be satisfactory. However, this same performance could not be expected during unit operation since the hot reactor is to be mounted inside the heat exchanger, thus causing performance degradation.

3.1.3 Compressor

A simple flow rate versus output pressure test was performed on the compressor assembly. The rated output of the carbon-vane compressor was 1.0 CFM, running open, to 0.8 CFM at 10 psig outlet pressure. Due to faulty manufacture of the carbon vane compressor, only 40% of this performance was attained. Modification of the carbon vane compressor in our shops increased the performance to 60% of rated output. A new carbon vane compressor and motor were obtained and fabricated into a modified compressor. Full output is obtained with this compressor assembly. While operating with 60°F inlet gas temperature, an outlet gas temperature of 120°F is obtained. No leakage of reaction gases are observed from the compressor assembly.

3.1.4   Electrolysis Unit

The multiple cell unit was placed in a test rig which included a means of feeding electrolyte and taking the gases off through a bubble trap.   After purging with argon, the unit was filled with 110 ml of 6N KOH, 6 ml less than the calculated amount. This was done 1.5 hours before the power was turned on.   However, this proved to be too short a time for proper soaking of the cells.   The initial soaking of the unit is important for proper operation, since an incomplete wetting will make the cell act as if it were dehydrated.   This, in fact, did happen and a small internal explosion was heard after the unit had been operating for five minutes at 10 amperes.   A check made on a spare wick revealed that the heating necessary to cure the epoxy adhesions was sufficient to grossly alter the initial wicking properties of the polypropylene.   As a result, the initial wetting was several times slower than might be expected.   To insure complete wetting, the cell was then partially flooded.   The excess electrolyte came over in the gas stream and the unit then operated well with no further electrolyte carry over.   Table I gives some of the operating data obtained for the unit during various runs.

TABLE I

ELECTROLYSIS UNIT OPERATION

Date	Operating Time	Cell Current	Average Voltage	Max. Cell Temp.
9-29-63	1.5 hr	50 amps	1.60	100°F
9-30-63	4.0 hr	50 amps	1.60	125°F
11-13-63	6.5 hr	65 amps	1.80	124°F
11-14-63	2.25 hr	65 amps	1.85	125°F
11-14-63	3.50 hr	65 amps	1.80	126°F
11-15-63	2.75 hr	65 amps	1.80	133°F
11-15-63	6.00 hr	65 amps	1.80	133°F

Total operating time = 26.5 hours

65 amps = 100 amps/sq ft

A run was made without adding feed water. When the unit was dehydrated, traces of hydrogen came over in the oxygen stream. When enough make-up water had been added, hydrogen was no longer detected. A properly hydrated cell does not allow hydrogen to come over to the oxygen side.

As the data show, the cell temperature seems to reach an equilibrium at about 125-130°F external temperature. A theoretical analysis shows that the bulk of the cell unit cooling is obtained by evaporative water losses during electrolysis. This water must be taken into account when calculating water consumption.

Figure 3-2 presents a curve giving the electrolysis unit voltage as a function of current density, as obtained from experimental data.

3.2 Unit Tests

Prior to endurance runs, preliminary tests are being carried out to determine the true operating characteristics of the unit. It is planned to determine the reaction rates obtainable as a function of reactor temperature, condenser-separator temperature, and recycle gas recirculation rate. During this preliminary testing phase, the electrolysis unit will be operated as a separate unit. The basic CO_2 reduction unit used in the testing is shown in Figures 3-3 through 3-7. Five views are shown of the unit just after completion of pressure checks prior to application of thermal insulation around reactor, heat exchanger, and filter assembly. Changes made in the unit since these photos were made will be included in the final report.

3.2.1 Experimental Run #1

Total running time was 130 minutes at an average reactor temperature of 1140°F, a condenser temperature of 70°F, and a reaction rate varying between 300-750 cc/min. The carbon blow-out cycle was tested, resulting in pressure build-up in the reactor and pressure decrease in the condenser-separator. Reaction water from the H_2O accumulator was drawn through the condenser-separator into the blower, causing complete system malfunction. The cause of the reactor blockage is not known. Unit overhaul commenced immediately.

AMPS/SQ. FT.
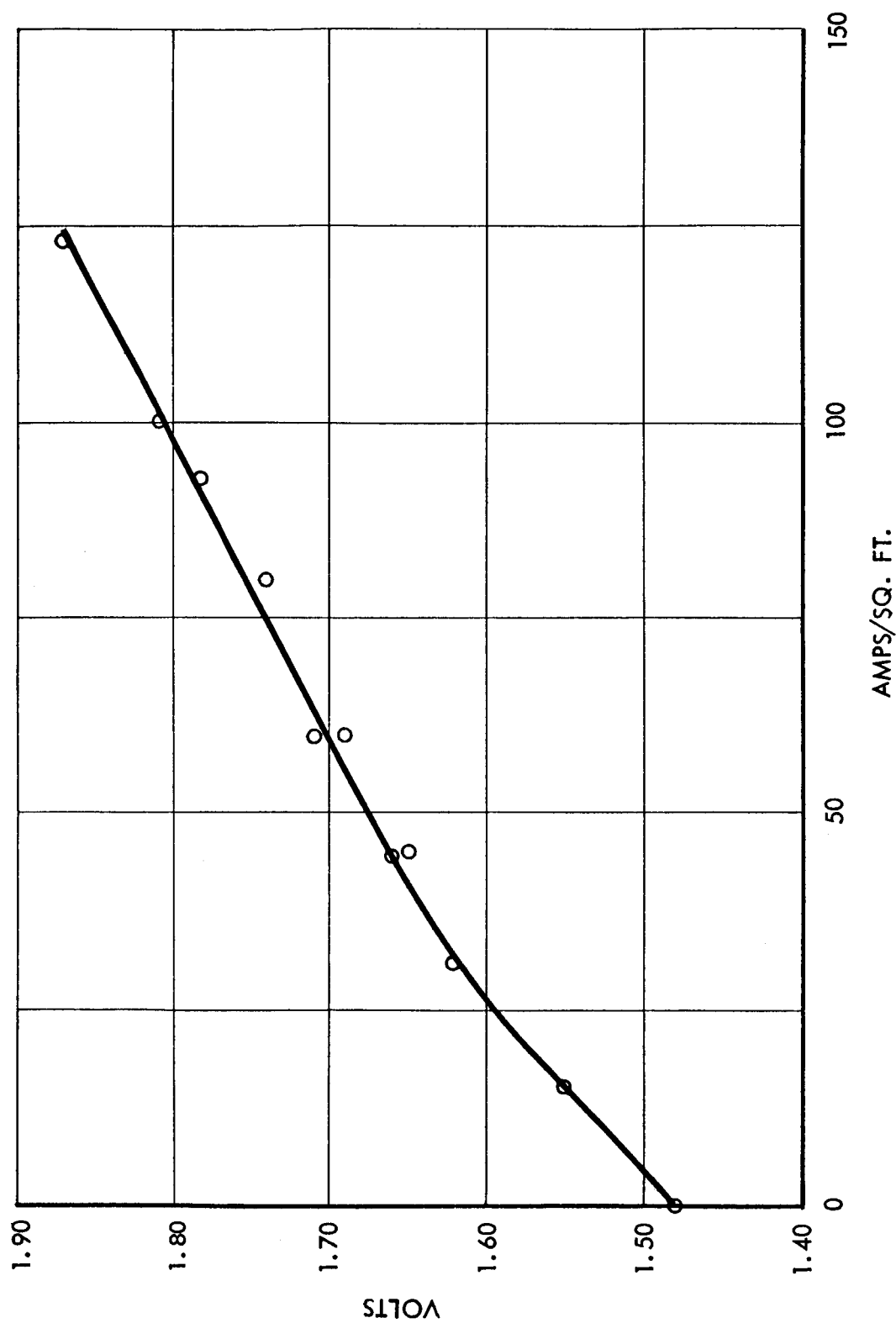
H$_2$O ELECTROLYSIS UNIT - VOLTAGE VS. CURRENT DENSITY
EXPERIMENTAL DATA (CELL TEMPERATURE = 125°F)

FIGURE 3-2

32

CO$_2$ REDUCTION UNIT – INSTRUMENT AND CONTROL PANEL

FIGURE 3-3

ELECTROLYSIS
UNIT

COMPRESSOR

CONDENSER
SEPARATOR

HEAT
EXCHANGER

PARTIALLY INSULATED
REACTOR
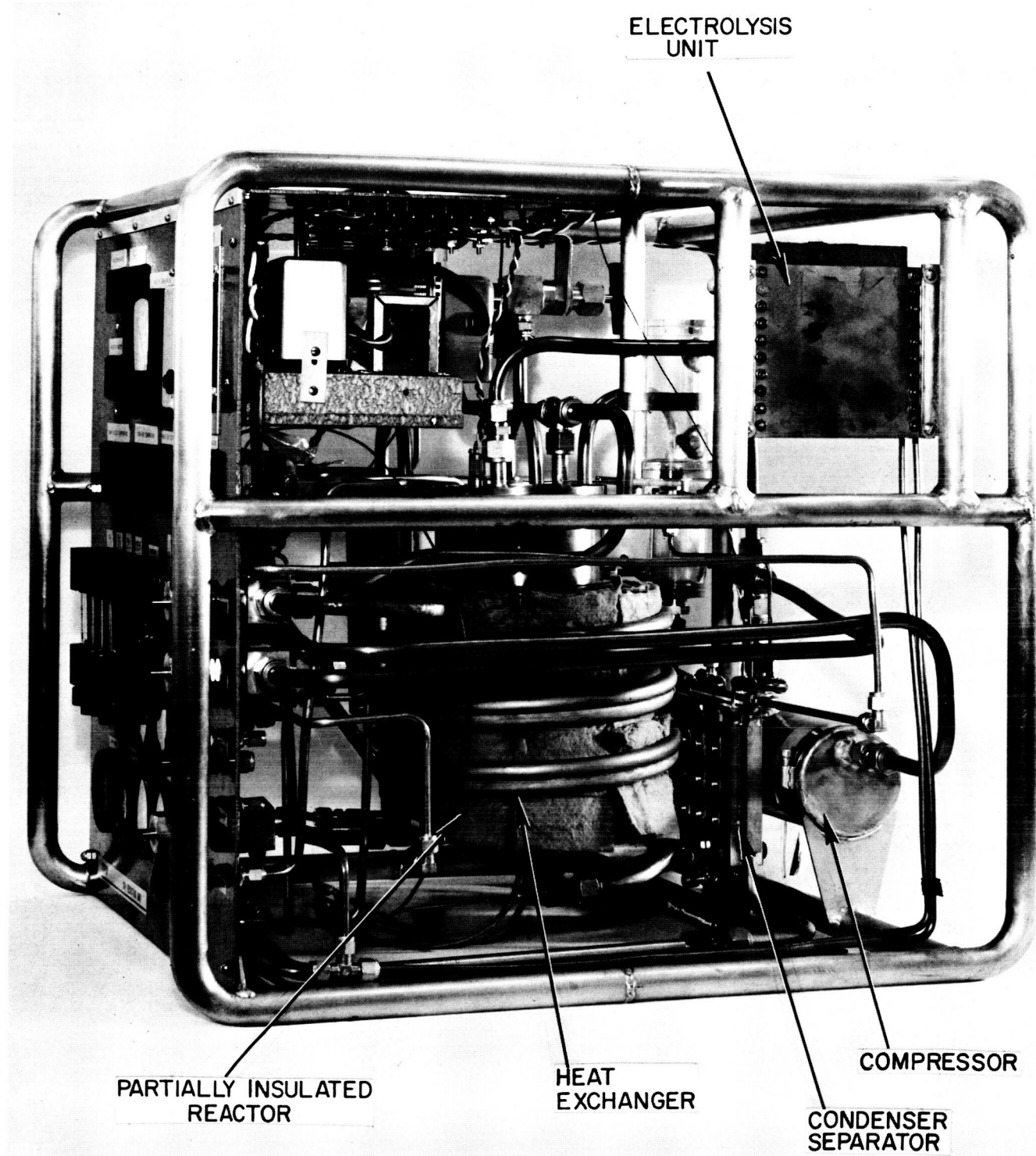
CO_2 REDUCTION UNIT – PARTIALLY ASSEMBLED

FIGURE 3-4
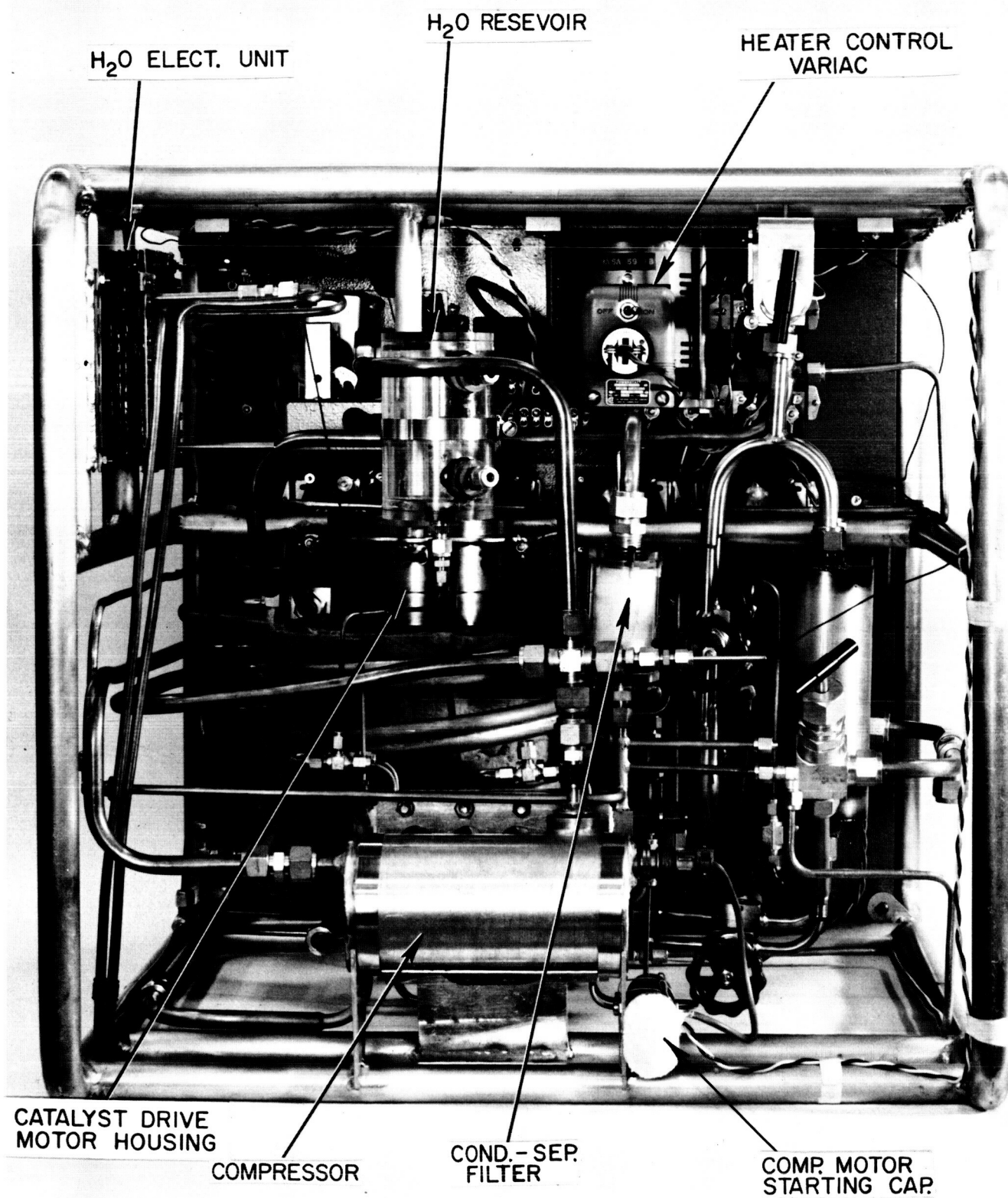
CO_2 REDUCTION UNIT – REAR VIEW

FIGURE 3-5

CARBON BLOW OUT
VALVES

FILTER SELECTOR
VALVES

POROUS STAINLESS
STEEL CARBON FILTERS

FILTER SELECTOR
VALVES
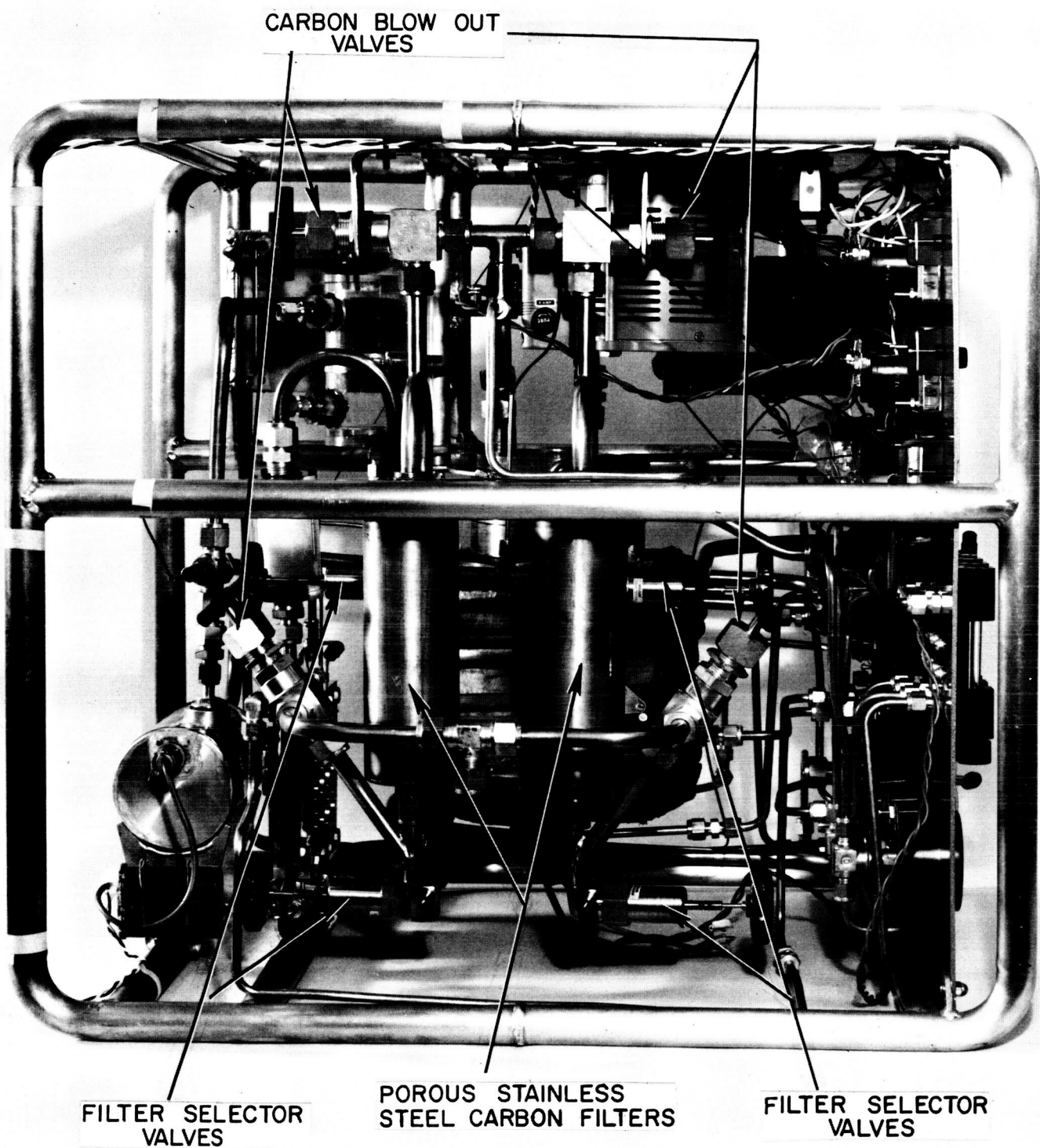
CO_2 REDUCTION UNIT – SIDE VIEW

FIGURE 3–6

36

CARBON FILTERS

CON.-SEP.
PRE-FILTER

H_2O ACCUMULATOR

CONDENSER
SEPARATOR

COMPRESSOR

H_2O ELECTROLYSIS
UNIT

REACTOR &
HEAT EX.
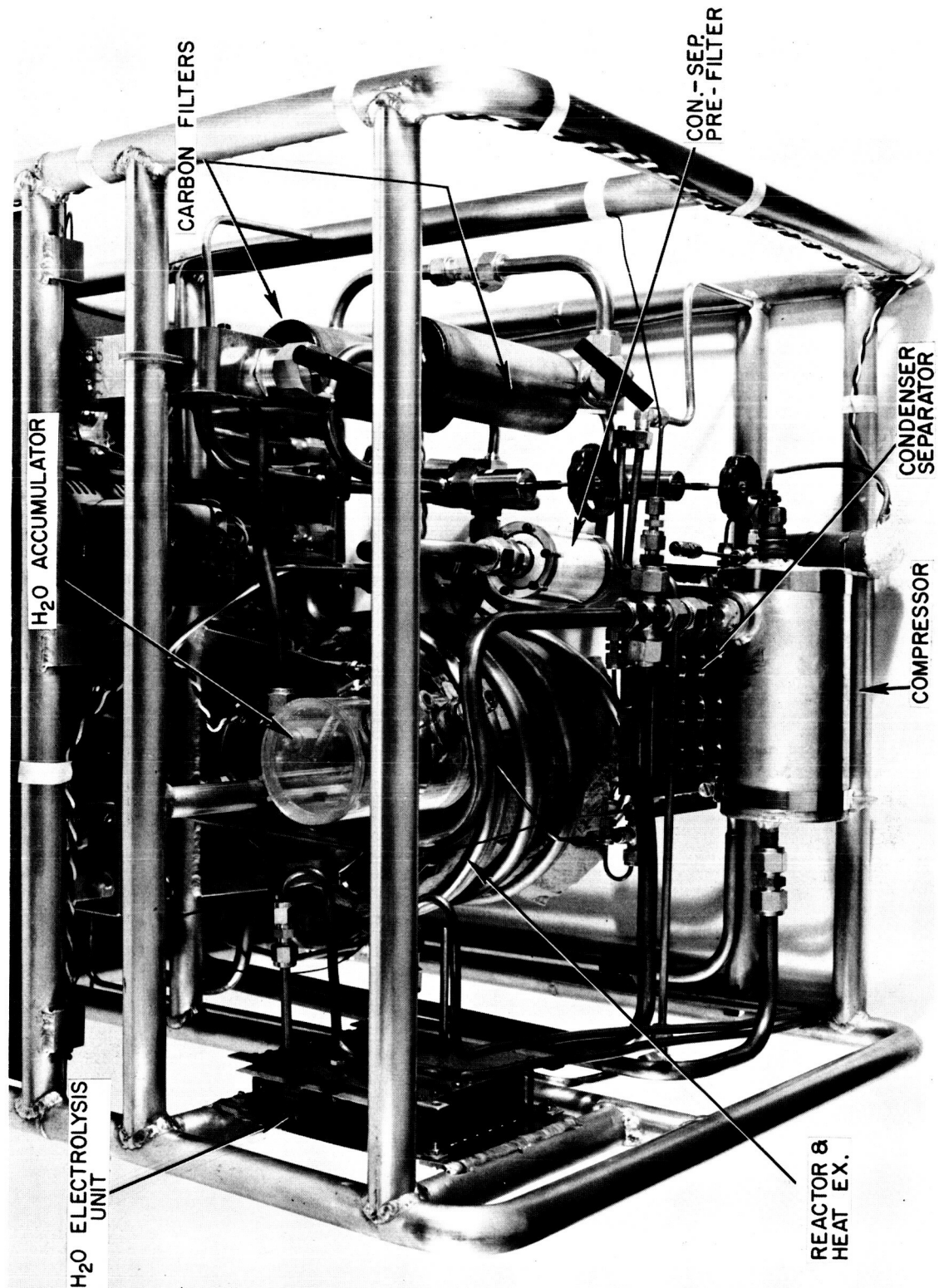
CO_2 REDUCTION UNIT – TOP VIEW

FIGURE 3-7

3.2.2 Experimental Run #2

A summary of results of experimental run #2 is given in Table II. This run was terminated after 27 hours 10 minutes running time to make changes in the unit as dictated by experimental observation. These changes were:

1. Replace compressor with modified compressor design.
2. Replace plastic filter prior to condenser-separator with all metal filter housing.
3. Install moisture trap in recycle flow line after condenser-separator.
4. Install new columns in gas chromatograph and recalibrate.
5. Change piping arrangement for carbon blow out cycle.

During run #2, the cause of the reactor blockage was determined as reaction water in the hot reaction gases condensing on the surface of the cold filter being switched into the cycle. The condensing water clogged the filter pores. This may be avoided by preheating both filters when the unit is first started. Thermal insulation will prevent filters from cooling to a temperature low enough to cause condensation.

The erratic reaction rate was the result of unsatisfactory gas analysis being obtained with the chromatograph. Thus the recycle gas composition varied over a wide range, giving rise to the varying reaction rate.

After the entire unit had cooled and flow through both filters was again possible, the carbon blow-out cycle was operated successfully, depositing carbon in the carbon collector.

TABLE II

EXPERIMENTAL RESULTS – RUN #2

Reactor Pressure = 2.0 psi Condenser Temp (Average) = 57°F

Date	Time	Feed rate cc/min	Recycle Flow Rate liters/min	Reactor Temp. °F	Heater Power Watts	Remarks
11-14-63	1030	–	–	1130	250	Begin feeding $2H_2:CO_2$ Gas Mixture
"	1115	490	15.5	1180	173	Additional H_2 Feed
"	1200	343	–	1095	173	Water in Recycle Rotameter
"	1400	186	16.3	1080	211	Reaction Gas Comp. out of Balance
"	1445	320	16.3	1145	226	
"	1500	451	16.3	1150	214	
"	1615	183	15.0	1130	196	
"	1645	366	16.8	1125	196	
"	1815	329	13.8	1280	289	
"	1915	400	14.6	1335	64	
"	2030	246	16.3	1180	245	
"	2200	350	14.6	1240	231	
"	2400	285	12.4	1230	231	Attempt to Switch Filters
11-15-63	0050	328	16.3	1220	250	
"	0145	441	14.6	1240	260	
"	0330	391	16.3	1220	308	
"	0415	405	18.8	1240	292	
"	0500	336	20.0	1240	308	
"	0635	398	18.0	1240	292	
"	0715	323	20.0	1245	292	
"	0830	308	19.0	1255	292	68 cc/min H_2 Feed
"	0930	416	19.0	1260	292	
"	1030	408	18.0	1260	292	
"	1130	422	18.0	1265	292	Gas Analysis – 1135 H_2-13.8%, N_2-10.6%, CH_4-24.1%, CO-39.5%, CO_2-12.0%
"	1200	440	18.0	1265	292	CH_4-35.5%, CO-21.5%, CO_2-18.5% H_2-14.6%,
"	1230	421	21.5	1265	292	N_2-11.5%
"	1300	384	18.0	1265	–	Heater Off – Prepare for Shutdown
"	1317	428	16.3	1070	–	
"	1325	381	18.0	970	–	
"	1332	250	18.0	910		
"	1340	198	18.0	845	–	

4.0 OBJECTIVES

It is anticipated that the following objectives will be attained as scheduled in TRW proposal, ER-5085.

1. Completion of preliminary unit testing to determine favorable operating conditions.

2. Completion of test period of six weeks total operating time for the CO_2 Reduction Unit.

3. Integration of CO_2 Reduction Unit with H_2O Electrolysis Unit into one operating system.

4. Operation of entire integrated system for three weeks of continuous running.

5. Demonstration of the feasibility of continuous carbon removal from a reduction system.

6. Demonstration of compact and efficient electrolysis unit.

7. Evolution of a program for future development of practical Integrated Oxygen Regeneration System for space usage.

5.0 RECOMMENDATIONS

Based on experimental results obtained to date and studies currently underway at TRW, the following program is recommended.

1. The present CO_2 Reduction Unit should be used as an investigative tool to gain more insight into the reduction of CO_2 by hydrogen with various catalyst materials under varying reaction conditions.

2. A multi-man flight prototype Integrated Oxygen Regeneration Unit should be designed, fabricated, and tested. This unit would include CO_2 Reduction with continuous carbon recovery, water electrolysis, and CO_2 concentration. As a means of increasing the reaction rate (decreasing electrical power consumption), a regenerable chemical recycle gas dryer would be included along with the present condenser-separator. Also a continuous bleed of reactor gases through a toxin burner would be included to remove nitrogen contaminant and to induce a more efficient reaction.

3. The present unit should be tested in a closed cabin to determine what, if any, toxic gases may be produced.

6.0   CONCLUSIONS

On the basis of experimental results obtained to date, the following conclusions are believed to be justified.

1. Construction of reactors using flat iron plates as catalyst is entirely feasible.

2. Continuous carbon removal from the plates is possible.

3. Porous stainless steel filters can be cleaned and used continuously.

4. Reaction water can be successfully removed in a zero-gravity environment by a completely static device.

5. Extremely compact and efficient water electrolysis units for zero-gravity operation can be built without the need of electrolyte circulation or cell rotation.

6. Satisfactory reaction rates can be continuously maintained by proper control of feed gases.

7. CO_2 reduction by H_2 over iron catalyst appears to be feasible for adaptation to space requirements.

REFERENCES

1. "Carbon Dioxide Reduction System," TRW Report No. ER-4351, February 17, 1961.

2. "Electrolytic CO_2 Reduction System," TRW Report No. ER-4701, December 15, 1961.

3. Foster, J. F., and J. S. McNutty, "Study of a Carbon Dioxide Reduction System," ASD TR 61-388, August, 1961.

4. Remus, G. A., R. B. Neveril, and J. D. Zeff, "Carbon Dioxide Reduction System," Report MR 1163-50, Contract No. AF 33(616)-8223, September, 1962.

5. "The Reduction of Carbon Dioxide Using a Fluidized Catalyst Bed," TRW Report No. ER-5159, Contract No. Nonr 3431(00)(FBM), December 15, 1962.